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Plasma Enhanced Chemical Vapor Deposition (PECVD) of Silicon Dioxide (SiO2) Using Oxford Instruments System 100 PECVD

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Abstract
This report discusses the deposition process of SiO2 using the Oxford System 100 PECVD.

Disciplines
Nanoscience and Nanotechnology

1. Introduction

This report documents the study of deposition characteristics and film properties of silicon dioxide (SiO_2) thin films deposited by plasma enhanced chemical vapor deposition (PECVD) using *Oxford PlasmaLab 100* system. Deposition rate, thickness non-uniformity, optical constant such as refractive index; in-plane stress and process repeatability were examined of SiO_2 films.

2. Tools and Techniques used

I. Oxford PlasmaLab 100 PECVD system was used for deposition of SiO_2 films on 100 mm (4-inch) <100> orientation Si wafers of thickness 525 ± 25 µm.
II. Filmetrics F50 optical interferometer was used for measuring the thickness of deposited films, non-uniformity in thickness over the wafer and optical constants.
III. Woollam VASE ellipsometer was used for measuring film thickness and optical constants.
IV. KLA Tencor P7 profilometer was used for measuring in-plane stress in SiO_2 films.

3. Baseline Recipe

Following baseline recipe was used for film deposition after loading the wafer in to the chamber via loadlock:

Units:

- Gas flow rate: standard cubic centimeters per minute (sccm)
- Pressure: millitorr (mT)
- Temperature: degrees Celsius (°C)
- High frequency (RF) and low frequency (LF) power: Watts (W)

Step 1: System chamber is pumped below 5 mT base pressure for 1 minute with electrode temperature at 350 °C.

Step 2: Chamber is pre-heated and purged with N_2 having flow rate of 700 sccm at pressure set point of 1400 mT and electrode temperature at 350 °C for 1 minute (for 4-inch wafer). *

*Step 2: If you are processing pieces mounted on a carrier substrate, it is recommended that the time in step 2 be increased to 10 minutes to ensure temperature stabilization of your samples.

Step 3: SiO_2 is deposited in this step with following precursors and chamber conditions:

- Silane (10 % SiH_4 in Helium) flow rate: 265 sccm
- Nitrous Oxide (N_2O) flow rate: 1000 sccm
- Nitrogen (N_2) flow rate: 500 sccm
- Pressure: 1800 mT
- High frequency RF power: 140 W
- Low frequency LF power: 0 W
- Capacitor starting points: Capacitor #1: 77 %, Capacitor #2: 26 %
- Electrode temperature: 350 °C
- Deposition time set point is hh:mm:ss (hours:minutes:seconds)

Step 4: Chamber is pumped to base pressure and wafer removed from loadlock.

4. Deposition Characteristics and film properties

The following sections will discuss the deposition characteristics and film properties on varying power (high frequency RF) and deposition time.

4.1 Deposition Rate

To study the effect of variation of power on deposition rate and film properties, 13 deposition runs were carried out with high frequency RF power in the range of 20 W to 260 W, with 20 W increment with each run. To rule out effect of process time to output, all 13 depositions were done for 1 minute. Figure 4.1 shows that the deposition rate increases with power (logarithmic variation) and saturates at about 220 W. Data used in figure 4.1 is presented in table 4.1 as measured by *Filmetrics F50*. The blue curve shows deposition rate attainable is in the range of 70 to 320 nm/min. The red curve shows standard deviation for each power. The black vertical bars at each point on the blue curve denotes the maximum and minimum SiO_2 deposition rate on the wafer. The deposition rate is calculated based on the average film thickness on the wafer as measured by *Filmetrics F50*. *Filmetrics F50* is equipped with a motorized stage allowing for the collection of full wafer maps as shown in figure 4.2 (wafer map of deposition run at 160 W for 1 minute). Thickness at 115 points per wafer was measured with 5 mm edge exclusion.

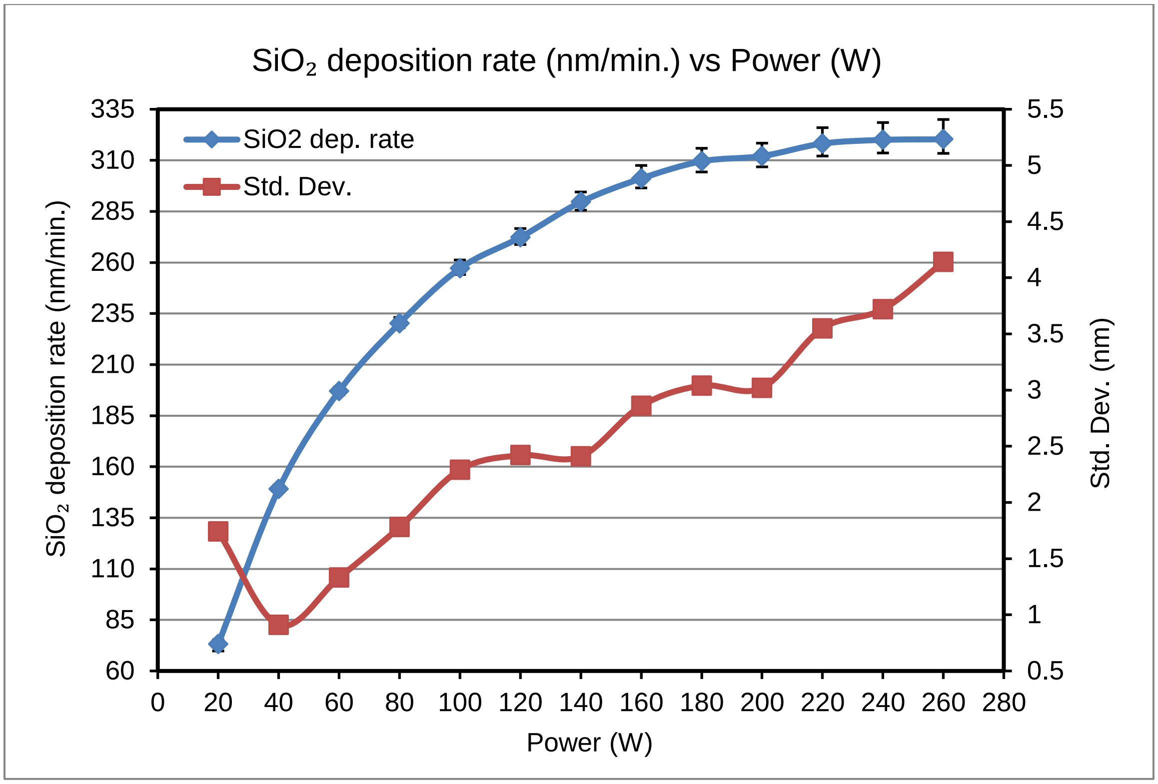


Figure 4.1: Variation of deposition rate with power.

Power (W)	Dep. Rate (nm/min.)	Avg. Thickness (nm)	Std. Dev. (nm)	Non-uniformity (%)
20	73.1	73.1	1.74	3.7
40	149	149	0.91	1
60	197	197	1.33	1
80	230.2	230.2	1.78	1.1
100	257.1	257.1	2.29	1.4
120	272.3	272.3	2.42	1.4
140	289.6	289.6	2.41	1.5
160	301.1	301.1	2.86	1.8
180	309.5	309.5	3.04	1.9
200	312.1	312.1	3.02	1.9
220	318.2	318.2	3.55	2.2
240	320	320	3.72	2.3
260	320.3	320.3	4.14	2.6

Table 4.1: Deposition rate and thickness non-uniformity as measured by Filmetrics F50.

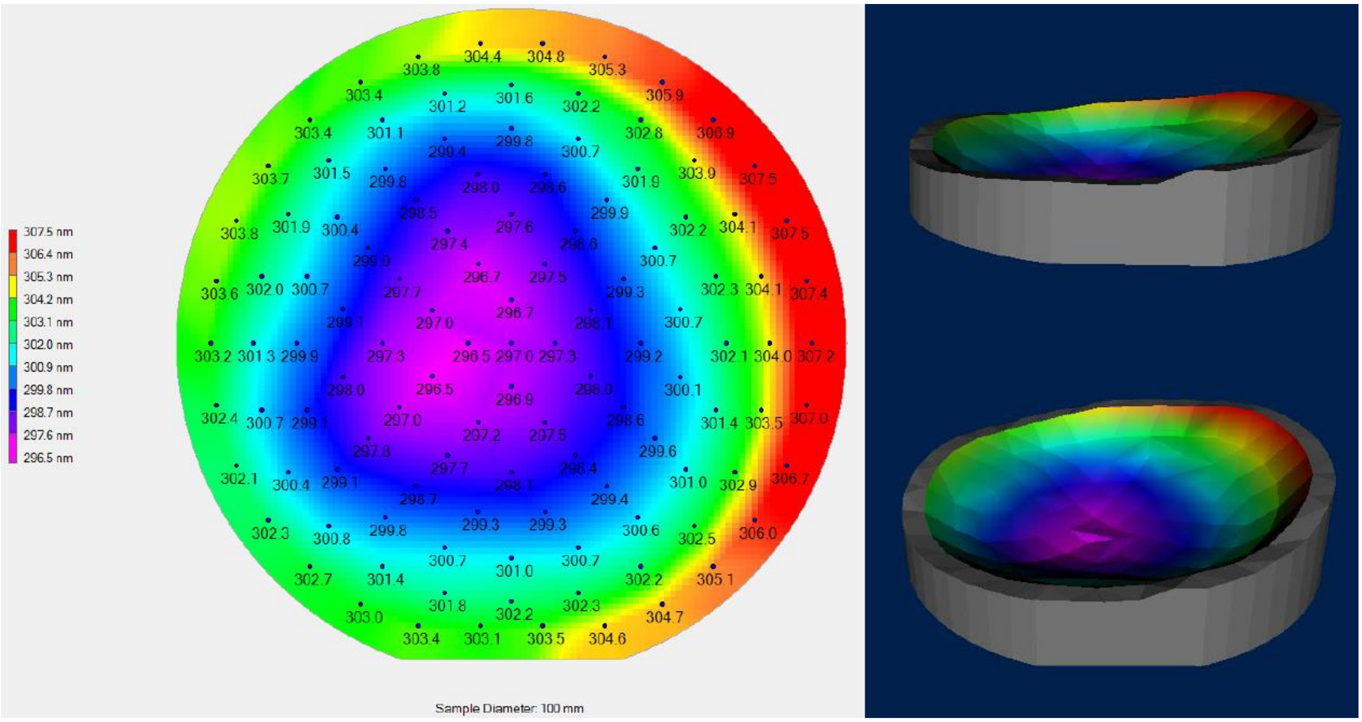


Figure 4.2: 2D map of thickness profile on the left, 3D maps on the right.

4.2 Thickness non-uniformity

Figure 4.3 shows the non-uniformity in film thickness as measured by *Filmetrics F50*. Except for the anomaly in non-uniformity for deposition done at 20 W, there is almost linear increase in non-uniformity in film thickness with increasing power. Data used in figure 4.3 is presented in table 4.1.

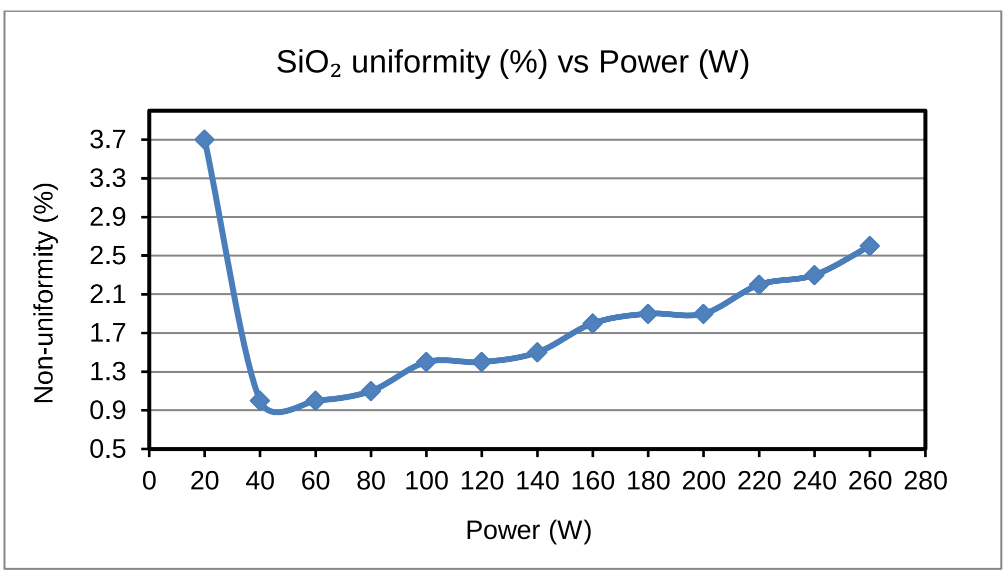


Figure 4.3: Thickness non-uniformity as measured by Filmetrics F50.

4.3 Repeatability of deposition rate and uniformity

To examine the repeatability and variation of deposition rate and thickness non-uniformity over multiple depositions for same power and deposition time, back to back depositions were done in two batches. Each batch consisted of 9 wafers. In the first batch, deposition was done at 160 W for 30 seconds. In the second batch, deposition was done at 160 W for 2 minutes. The results from two batches were compared to examine the variation in deposition rate for same time as well as different deposition time.

<u>Batch 1: 9 back to back depositions, 160 W power, 30 seconds of deposition time each</u>

Figure 4.4 shows the average thickness and its standard deviation measured by *Filmetrics F50* in 9 wafers where deposition was carried out for 30 seconds. From the above thickness, the average deposition rate for the 9 samples was 305.72 nm/min. with standard deviation of 1.64 nm/min. Figure 4.5 shows thickness non-uniformity in the 9 wafers. There is variation in non-uniformity with back to back depositions, average being 2.52 % with standard deviation of 0.29 %. Data used in figures 4.4 and 4.5 is presented in table 4.2.

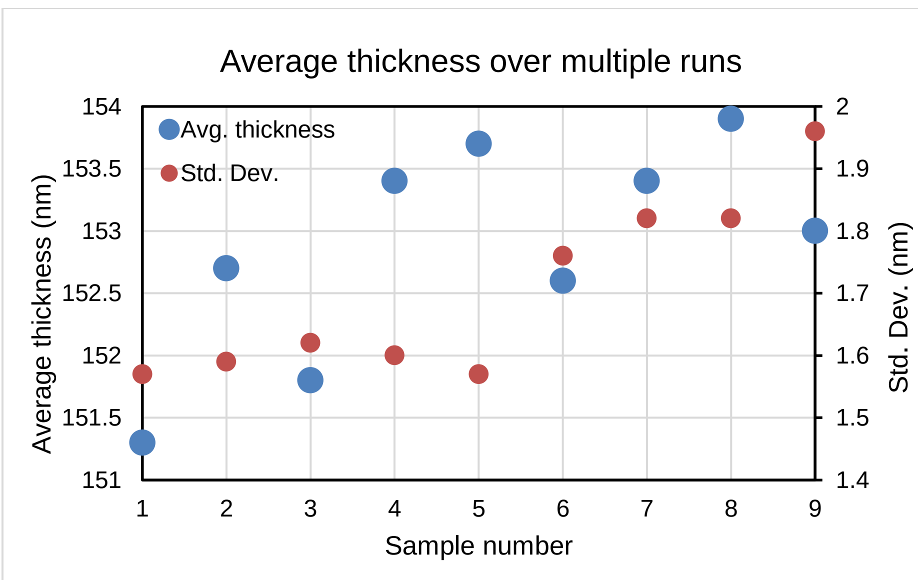


Figure 4.4: Average thickness in multiple runs of 30 seconds each as measured by Filmetrics F50.

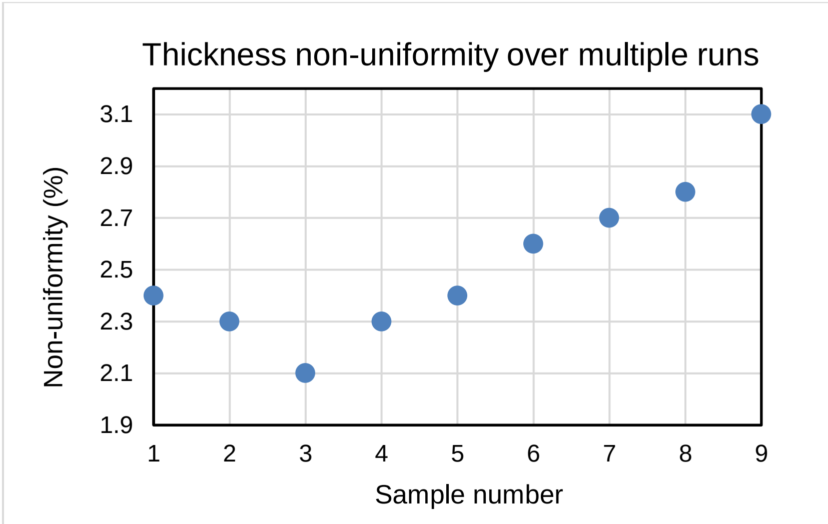


Figure 4.5: Thickness non-uniformity in multiple runs of 30 seconds each as measured by Filmetrics F50.

Batch	Sample	Avg. Thickness (nm)	Std. Dev. (nm)	Non-uniformity (%)	Refractive index 'n'
1	1	151.3	1.57	2.4	1.4716
	2	152.7	1.59	2.3	-
	3	151.8	1.62	2.1	1.4712
	4	153.4	1.6	2.3	1.4713
	5	153.7	1.57	2.4	1.4712
	6	152.6	1.76	2.6	1.4714
	7	153.4	1.82	2.7	1.4712
	8	153.9	1.82	2.8	1.4710
	9	153	1.96	3.1	1.4714

Table 4.2: Batch 1 repeatability measurements. Average thickness, standard deviation and non-uniformity measured by Filmetrics F50. Refractive indices measured by Woollam VASE.

Batch 2: 9 back to back depositions, 160 W power, 2 minutes of deposition time each

Figure 4.6 shows the average thickness and its standard deviation measured by *Filmetrics F50* in 9 wafers where deposition was carried out for 2 minutes. From the above thickness, the average deposition rate for the 9 samples was 300.71 nm/min. with standard deviation of 1.98 nm/min. Figure 4.7 shows thickness non-uniformity in the 9 wafers. There is variation in non-uniformity with back to back depositions, average being 2.02 % with standard deviation of 0.1 %. Data used in figures 4.6 and 4.7 is presented in table 4.3.

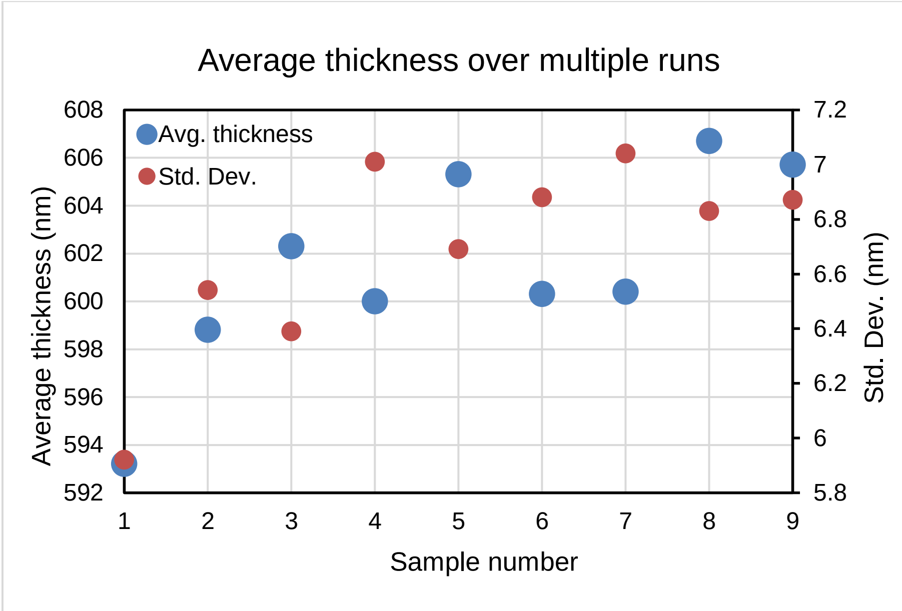


Figure 4.6: Average thickness in multiple runs of 2 minutes each as measured by Filmetrics F50.

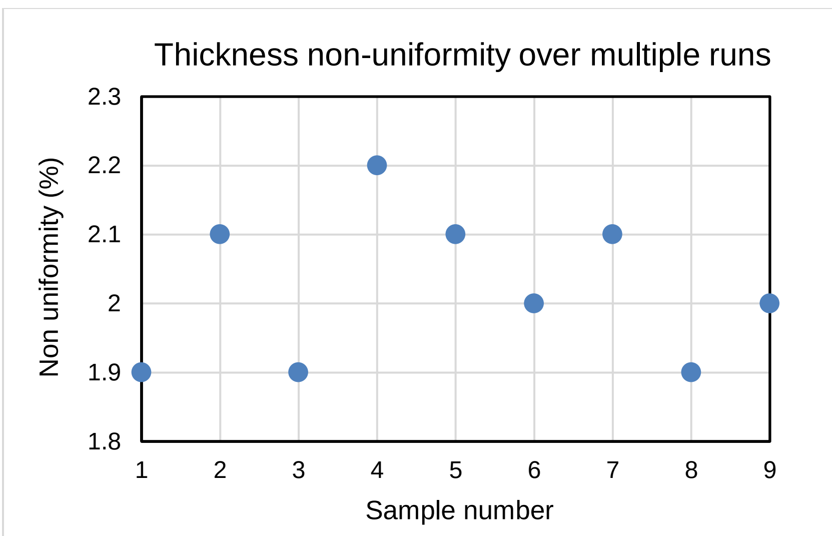


Figure 4.7: Thickness non-uniformity in multiple runs of 2 minutes each as measured by Filmetrics F50.

Batch	Sample	Avg. Thickness (nm)	Std. Dev. (nm)	Non-uniformity (%)	Refractive index 'n'
	1	593.2	5.92	1.9	1.4719
	2	598.8	6.54	2.1	1.4733
	3	602.3	6.39	1.9	1.4722
	4	600	7.01	2.2	1.4725

2	5	605.3	6.69	2.1	1.4726
	6	600.3	6.88	2	1.4725
	7	600.4	7.04	2.1	1.4728
	8	606.7	6.83	1.9	1.4727
	9	605.7	6.87	2	1.4725

Table 4.3: Batch 2 repeatability measurements. Average thickness, standard deviation and non-uniformity measured by Filmetrics F50. Refractive indices measured by Woollam VASE.

Table 4.4 summarizes the deposition rate and non-uniformity for different cases discussed above. For lower deposition time, the average deposition rate is slightly higher and average non-uniformity in thickness across the wafer is about 25% more. Thus, based on this data as well as figure 4.2, lower power should be used for thickness typically less than the deposition rate at respective power to get better uniformity across the wafer.

Conditions	Average deposition rate (nm/min.)	Average non-uniformity (%)
Single run, 160 W, 1 minute	301.1	1.8
9 runs, 160 W, 30 seconds	305.72	2.52
9 runs, 160 W, 2 minutes	300.71	2.02

Table 4.4: Summary of deposition rates and non-uniformity.

4.4 Optical Constants

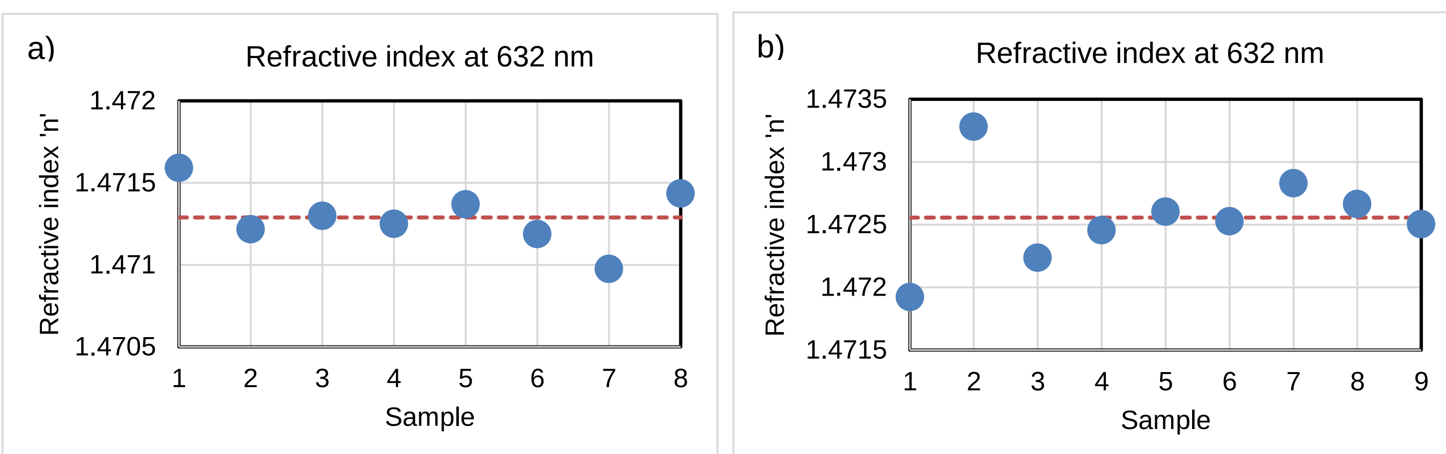


Figure 4.8: a) Refractive index 'n' from multiple runs of 30 seconds each. b) Refractive index 'n' from multiple runs of 2 minutes each. Orange line denotes mean 'n'. Refractive indices are as calculated from Woollam VASE ellipsometer measurements.

The refractive index 'n' of the samples from 160 W depositions is measured by ellipsometry technique to study the consistency in optical quality of the film among multiple depositions. *Woollam VASE* ellipsometer is used which measures thickness as well as optical constants. A quick spectroscopic scan is done between light wavelength of 240 nm to 1100 nm (with 20 nm increment in wavelength) at angles of 65° and 75°. SiO_2 film is modeled as a transparent film on Si substrate by using Cauchy relationship:

$$n(\lambda) = A + \frac{B}{\lambda^2} + \frac{C}{\lambda^4}$$

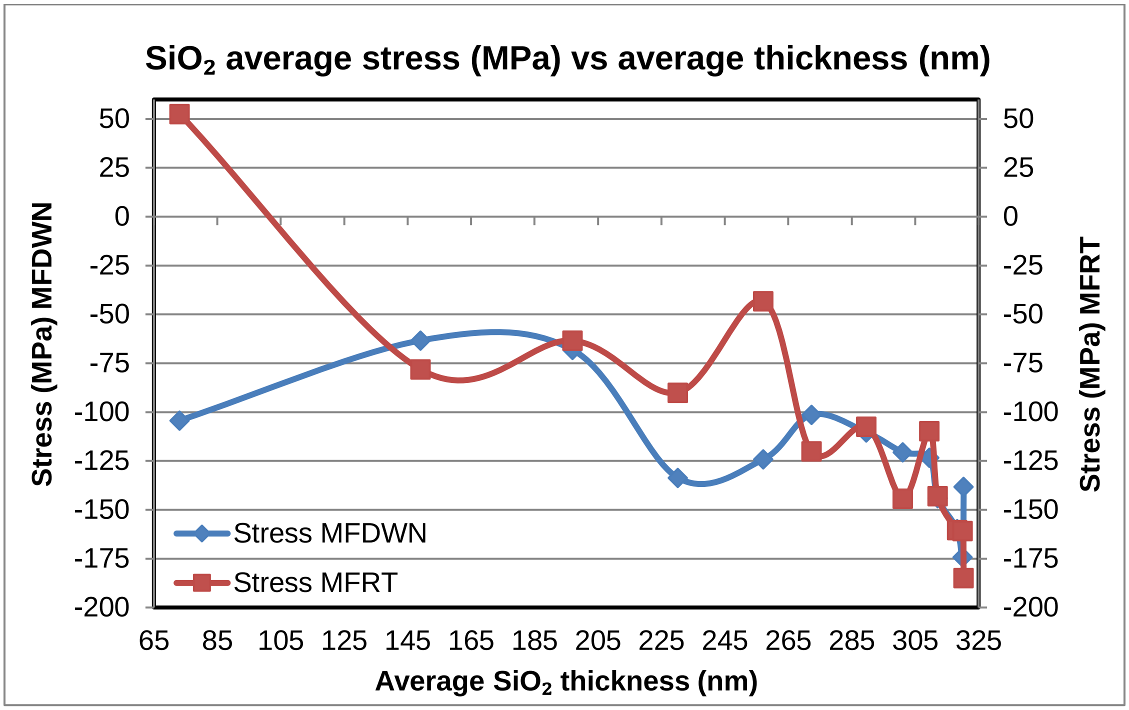
From the spectroscopic scan, experimental A and B values are determined (C is negligible) for each sample and 'n' calculated at 632 nm using the Cauchy relationship. Figure 4.8 a) shows calculated 'n' from experimental data for 8 samples deposited at 160 W for 30 seconds (one sample not included due to measurement error) and figure 4.8 b) shows 'n' for 9 samples deposited at 160 W for 2 minutes. The orange dotted line represents the mean 'n' for each set of samples: 1.4713 in a) and 1.4725 in b). The standard deviation of 'n' in a) is 0.000171 and in b) is 0.000355. Overall, the optical quality of the film is consistent among multiple depositions of same as well as different run times. Data used in figure 4.8 a) is presented in table 4.2 and that in figure 4.8 b) is presented in table 4.5.

Batch	Sample	Refractive index 'n'	Batch	Sample	Refractive index 'n'
1	1	1.4716	2	1	1.4719
	2	1.4712		2	1.4733
	3	1.4713		3	1.4722
	4	1.4712		4	1.4725
	5	1.4714		5	1.4726
	6	1.4712		6	1.4725
	7	1.4710		7	1.4728
	8	1.4714		8	1.4727
				9	1.4725

Table 4.5: Batch 1 and 2 refractive indices as calculated from Woollam VASE ellipsometer measurements.

4.5 Mechanical Stress

In-plane stress is measured to study the effect of deposition power on film stress. To measure in-plane stress, 2D stress measurement option in *KLA Tencor P7* profilometer is used. Film stress is measured in two perpendicular directions in center: one (MFDWN) parallel to the major flat axis of the substrate (MFDWN) and second (MFRT) perpendicular to the major flat axis of the substrate as shown on the right in figure 4.9.



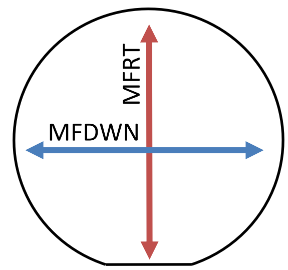


Figure 4.9: Variation of in-plane stress in SiO_2 films as measured by KLA Tencor P7.

Power (W)	Avg. Thickness (nm)	Stress (MPa) MFDWN	Stress (MPa) MFRT
20	73.1	-104.3	52.64
40	149	-63.38	-78.04
60	197	-68.17	-63.45
80	230.2	-133.6	-89.99
100	257.1	-124.2	-43.29
120	272.3	-101.3	-120.1
140	289.6	-110.3	-107.4
160	301.1	-120.5	-144.2
180	309.5	-123.3	-109.8
200	312.1	-144	-143
220	318.2	-159.9	-160.3
240	320	-174.2	-160.8
260	320.3	-138.2	-184.9

Table 4.6: Stress measurements using KLA Tencor P7.

Before depositing SiO_2 film, radius of curvature of the Si substrate is measured using the 2D stress option. After the deposition, radius of curvature of the deposited film is measured. The software in *P7* calculates the stress using the pre- and post-deposition radius of curvature and the input film thickness. The average film thickness as measured by *Filmetrics F50* is used to calculate stress. Since the stress calculation uses average thickness and does not consider the non-uniformity, stress calculated is approximate. Figure 4.9 shows the stress across MFDWN and MFRT for various thicknesses (corresponding to power in figure 4.1). Data used in figure 4.9 is presented in table 4.6. The film stress is compressive in nature and anisotropic in some cases (different stress across MFDWN and MFRT) whereas isotropic (similar stress across MFDWN and MFRT) in some cases.

Figure 4.10 shows the in-plane stress as measured in 9 samples deposited at 160 W for 30 seconds each. Data used in figure 4.10 is presented in table 4.7. Even with the same deposition conditions, stress is seen to vary among the samples. Except for one data point, rest indicates compressive nature of stress in SiO_2 films across both MFDWN and MFRT. Figure 4.11 shows the in-plane stress as measured in 9 samples deposited at 160 W for 2 minutes each. Data used in figure 4.11 is presented in table 4.8. Contrary to figure 4.10, all the data points indicate compressive stress and most samples shows similar stress across MFDWN and MFRT. The similar stress across the two axes indicate highly isotropic film. The results indicate that higher thickness films (~600 nm in 2 minute depositions compared to ~150 nm in 30 second depositions) tend to have isotropic compressive in-plane stress.

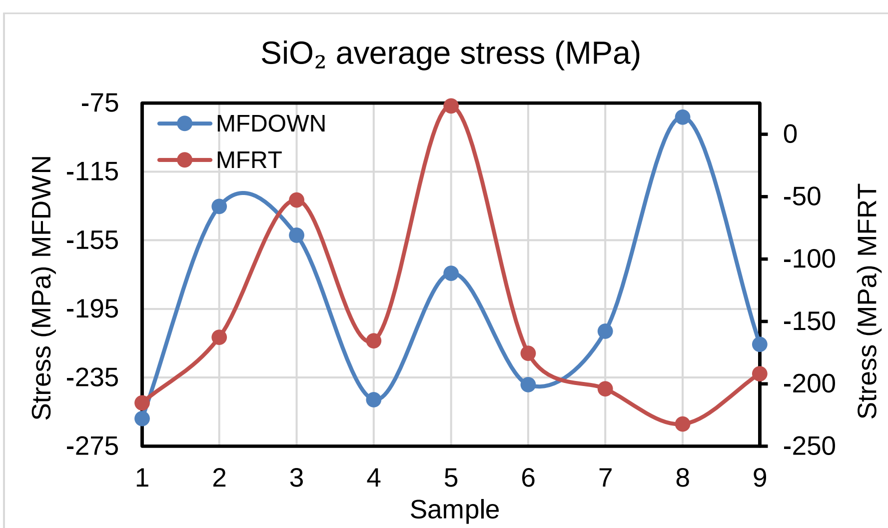


Figure 4.10: In-plane stress in SiO_2 films from multiple runs of 30 seconds each as measured by KLA Tencor P7.

Batch	Sample	Stress (MPa) MFDWN	Stress (MPa) MFRT
1	1	-258.8	-215.1
	2	-135.2	-162.6
	3	-151.9	-52.58
	4	-247.8	-165.4
	5	-174.1	22.74
	6	-239.1	-175.3
	7	-207.9	-204
	8	-83.07	-232.2
	9	-215.5	-191.7

Table 4.7: Batch 1 in-plane stress as measured by KLA Tencor P7.

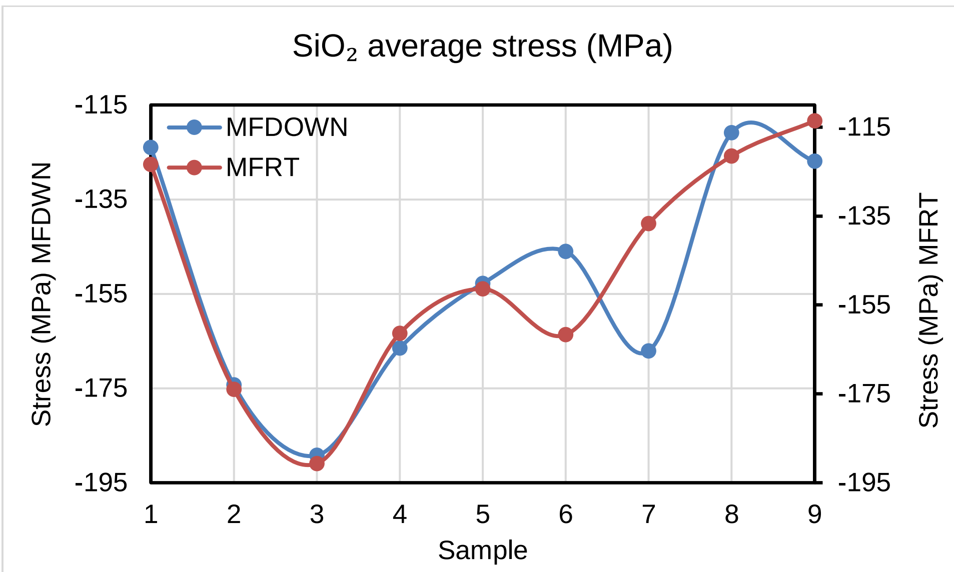


Figure 4.11: In-plane stress in SiO_2 films from multiple runs of 2 minutes each as measured by KLA Tencor P7.

Batch	Sample	Stress (MPa) MFDWN	Stress (MPa) MFRT
2	1	-124	-123.4
	2	-174.3	-174
	3	-189.2	-190.7
	4	-166.5	-161.4
	5	-152.8	-151.4
	6	-146	-161.7
	7	-167.1	-136.7
	8	-120.9	-121.5
	9	-126.9	-113.6

Table 4.8: Batch 2 in-plane stress as measured by KLA Tencor P7.

5. Summary

Deposition rate and film properties for SiO_2 films deposited by PECVD using *Oxford PlasmaLab 100* are examined. Tools such as *Filmetrics F50* (interferometer), *Woollam VASE* (ellipsometer) and *KLA Tencor P7* (profilometer) are used to measure thickness, optical properties and film stress. To examine effect of power on deposition rate, PECVD is carried out for varying power as shown in figure 4.1. The deposition rate is calculated using thickness measurement obtained by *F50*. The thickness measurement also provides information on non-uniformity of film thickness across the wafer. Further, multiple (18) depositions were carried out at same power (160 W) in 2 batches: 9 depositions of 30 seconds each and 9 depositions of 2 minutes each. The data obtained from the 2 batches is analyzed to examine the process repeatability and variation of deposition rate, non-uniformity, refractive index ‘n’ and in-plane stress. Average deposition rate and non-uniformity is higher in 30 second depositions compared to 2-minute depositions whereas average refractive index ‘n’ is lower. 2 minute depositions result in isotropic compressive films compared to that in 30 second depositions. Overall, variation of deposition rate among the 2 batches is very small (~5 nm/min.) and similar refractive indices indicate consistency in optical quality of the deposited films.